



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification<sup>4</sup> :</b> <b>A61L 15/00, A61F 13/18</b> <b>A41B 13/02</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 89/ 06142</b> <b>(43) International Publication Date:</b> 13 July 1989 (13.07.89)
<b>(21) International Application Number:</b> PCT/GB89/00013 <b>(22) International Filing Date:</b> 3 January 1989 (03.01.89) <b>(31) Priority Application Numbers:</b> 8800093 8812632.1 <b>(32) Priority Dates:</b> 5 January 1988 (05.01.88) 27 May 1988 (27.05.88) <b>(33) Priority Country:</b> GB <b>(71)(72) Applicant and Inventor:</b> FORDE, Liam, Patrick [GB/GB]; 6 Knocklayde Park, Coleraine, Londonderry (GB). <b>(74) Agent:</b> FITZPATRICKS; 4 West Regent Street, Glasgow G2 1RS (GB).		<b>(81) Designated States:</b> AT (European patent), BE (European patent), CH (European patent), DE (European patent), FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), NO, SE (European patent), US.  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> pH CONTROLLING ABSORBENT PRODUCT AND METHOD FOR PREPARING THE SAME  <b>(57) Abstract</b>  In a diaper the absorbent material is acidic and capable of releasing H <sup>+</sup> ions to lower the pH of urine in contact therewith to below that in which bacteria producing ammonia can grow (about 5.5). The material can be naturally acidic such as sphagnum moss or peat but preferably a neutral absorbent material is protonated.		

***FOR THE PURPOSES OF INFORMATION ONLY***

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	ML	Mali
AU	Australia	GA	Gabon	MR	Mauritania
BB	Barbados	GB	United Kingdom	MW	Malawi
BE	Belgium	HU	Hungary	NL	Netherlands
BG	Bulgaria	IT	Italy	NO	Norway
BJ	Benin	JP	Japan	RO	Romania
BR	Brazil	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	LI	Liechtenstein	SN	Senegal
CH	Switzerland	LK	Sri Lanka	SU	Soviet Union
CM	Cameroon	LU	Luxembourg	TD	Chad
DE	Germany, Federal Republic of	MC	Monaco	TG	Togo
DK	Denmark	MG	Madagascar	US	United States of America
FI	Finland				

PH CONTROLLING ABSORBENT PRODUCT AND METHOD  
FOR PREPARING THE SAME

This invention relates to diapers, incontinent pads or sheets, and other sanitary products which come in contact with urine or the like, in which undesirable bacteria can grow.

Various types of absorbent materials such as wood pulp, starches, towelling, absorbent powders and synthetic polymers are used at present to absorb babies' urine and urea of incontinent and incapacitated persons in items such as diapers and bed sheets.

One-piece disposable diapers comprise, in order, a liquid-permeable or semi-permeable material adapted to be in contact with the skin, a relatively thick absorbent substrate, and an outer layer of an impermeable material, the whole suitably shaped and usually including means for fastening the nappy about the baby's waist. Relatively "old-fashioned" towelling diapers provide an absorbent substrate, and are often used together with, on the inside, a separate layer of a liquid-permeable or semi-permeable material, a separate pair of impermeable pants, and separate fastening means such as a safety pin. The general arrangement of materials is the same in both cases.

It is known that bacteria can grow in urine above a pH of about 5.5 and metabolise the urea in the urine, in the process producing ammonia. The ammonia in contact with the skin, can cause 'nappy-rash', or with incontinent people, bed-sores. Many attempts have been made to combat this problem, such as by using acidic compounds to capture or neutralise the ammonia, or lowering the pH of the urine to below 5.5 to inhibit the growth of the ammonia-producing bacteria.

A typical way in which these acidic compounds are added to the diaper is described in patent nos. GE 1484501, US 424242, US 4273786, US 3964486 and US 3920015. In these patents, the acidic compound is admixed with or lies powdered on the surface of the absorbent material, and dissolves when contacted with the urine. It is, however, possible to have areas of

absorbent or infill material which is not treated, and there is also a tendency with powders to concentrate in particular areas, for example the outer edge of the diaper, leading to variations in the pH of the urine such  
5 as acid deficient spots where the bacteria can still grow, or highly acidic spots which can cause discomfort to the baby.

It is an object of the present invention to obviate or mitigate the aforesaid disadvantages.

10 According to the first aspect of the present invention there is provided in a diaper, incontinent bedding and the like, the absorbent material of which is capable of releasing hydrogen ions to maintain the pH of urine in contact therewith to between 2 and 5.5 for a practicable period

15 The absorbent material itself is now acidic rather than having for example an acidic powder on the surface thereof. Therefore  $H^+$  ions can be released uniformly throughout the contacting urine mitigating the effect of pH variances. Furthermore final pH of the urine is more  
20 predictable and controllable. The invention may further aid in the absorbancy quality of the acidic material.

Preferably the  $H^+$  ions are carried by a hydroxyl base, and more preferably the absorbent material is a polymer of glucose.

25 The limits of the desired urine pH are defined by pH value below which the bacteria producing the ammonia cannot survive (about 5.5 and below); and by the pH value where the acidic urine causes acid burns or otherwise substantially irritates the skin (below about pH2).

30 Preferably the invention works in the pH range of 2.5 to 3. Obviously, however, because of the specificity that can be obtained, the absorbent material could be designed to give most desired urine pH values, such as for example, that required to capture the ammonia.

35 The acidic absorbent material can be admixed with other non-acidic absorbent materials, and naturally acidic or more preferably is substantially an absorbent material which has been protonated since it would not otherwise be sufficiently acidic.

Naturally acidic materials preferably include suitably sterilised sphagnum moss or peat (dead sphagnum moss). Both of these materials are also highly absorbent and sphagnum moss contains phenolic acid which has antiseptic qualities and lanolin which has soothing qualities. It should be noted, however, that sphagnum moss can contain, for example, heavy metals and, the moss should therefore be treated and sterilised to render the moss suitable for use. Preferably the natural material such as the sphagnum moss and peat are bleached, which sterilises, makes the material more attractive and does not substantially affect its acidic character. The sphagnum moss or peat can be mixed with a relatively neutral material such as wood pulp at, at least 10%, preferably 20% to 30% by weight of the moss or peat.

A high degree of pH control can be obtained if an insufficiently acidic absorbent material is protonated. Most absorbent materials in use are polymers of glucose, such as cellulose or starch based materials, and the free hydroxyl groups can act as bases. The degree of protonation and thus final urine pH will depend, for example, on the concentration and time of exposure of the  $H^+$  ions, as well as the strength of the base.

Preferably the absorbent material is soaked in an aqueous solution of any suitable acid, of pH for example from 1.8 to 6.5 and preferably from 3.5 to 6. Advantageously sulphuric or hydrochloric acid is used.

The process of protonating an absorbent material forms a second aspect of the invention.

According to second aspect of the invention, there is provided, a process of preparing an acidic absorbent material for a diaper, incontinent bedding or the like, comprising protonating an absorbent material capable of taking up hydrogen ions and of maintaining the pH of urine in contact therewith to from 2 to 5.5 for a practicable period.

The acidic absorbent material as defined in the first aspect, and made in accordance with the second aspect of the invention, forms the third aspect of the present invention.

The invention will now be illustrated by way of the following examples.

Series 1

Samples of sphagnum moss and peat were bleached.  
5 Each of these, and a sample of non-acid wood pulp were then contacted with urine under simulated body conditions and incubated for some time. Two sets of tests were run (see tables I and II) and in each case the sample weight used was 5g and the weight of fresh urine was 27g. In  
10 the second run (table II) the results were taken over several days.

It is well known that above about pH 5.5, gram negative and gram positive bacteria will grow with the accompanying generation of ammonia.

15 No ammonia ( $\text{NH}_4$ ) was detected in the sphagnum moss or peat samples, whereas with the wood pulp, ammonia was detected after 6 hrs in the first sample (urine pH 5.3) and after 3/4 hr in the second sample (urine pH 6.6).

Series 2

20 A typical commercial product, i.e. a baby's diaper as sold by a supermarket, was taken and the absorbent infill was removed and examined.

It was found that the infill product was wood pulp, and this was weighed to record its dry weight. Various  
25 solutions of dilute sulphuric acid were prepared having the following pH values.

Sample No. 1 pH 2.00

Sample No. 2 pH 3.10

Sample No. 3 pH 3.80

30 Sample No. 4 pH 6.50

The wood pulp infill was divided into eight equal parts and two parts were used with each of the sample acids.

The first of the two portions of wood pulp was completely submerged in the dilute sulphuric acid for a  
35 period of 30 seconds. It was found that during this 30 seconds the wood pulp soaked up dilute acid and became completely and totally saturated. The second of the two portions of wood pulp was allowed to remain in the dilute acid for a period of up to three hours, at which stage it

was completely saturated. This was repeated for each of the other three acid solutions.

On removal from the dilute acid the wood pulp was squeezed to remove as much as possible of excess acid, and the sample was then dried at 102°C, until it was found to be 100% dry. The sample was then weighed and its weight checked against the original dry weight.

Eight samples of wood pulp infill from a commercial diaper were prepared in this manner.

A properly calibrated pH meter and pH paper were both used in the following tests on the treated samples.

A concentrated solution of ammonia pH 10.4 was used to test the acidic qualities of the treated samples. Ordinary water pH 7.8 was also used to determine the acidic qualities of the treated samples.

The following results were achieved using wood pulp samples which had been immersed.

Sample No. 1 (in dilute acid for only 30 seconds).

Acid pH 2.00

Ammonia pH 10.4 reduced to pH 8.2

Water pH 7.8 reduced to pH 2.8

Sample No. 2

Acid pH 3.4

Ammonia pH 10.4 reduced to pH 8.9

Water pH 7.8 reduced to pH 4.0

Sample No. 3

Acid pH 3.8

Ammonia pH 10.4 reduced to pH 9.1

Water pH 7.8 reduced to pH 4.8

Sample No. 4

Acid pH 6.5

Ammonia pH 10.4 reduced to pH 10.0

Water pH 7.8 reduced to pH 7.2

These tests were then repeated on the wood pulp samples which had been immersed in dilute acid for a period of three hours and the same results were obtained in all cases.

The reason that the wood pulp became protonated was because starch and cellulose are polyers of glucose. The

glucose molecule has three free OH groups. The hydrogen ions from the acid attach themselves to the oxygen of the hydroxyl group forming an ionic compound.

Whenever water enters the glucose molecule the hydrogen ions prefixed to the OH group are released and combine immediately with the hydrogen in the water molecule forming  $H_2O$ . Any sulphate ions present are in a very stable condition and are thus harmless.

The concentration of hydrogen ions in the infill material can be varied by submerging it in different strengths of the dilute acid. By this means, a controlled neutralisation process can be achieved. The last wash of the wood pulp at the wood pulp factory, prior to drying, can be carried out in a dilute acid solution, to suit a ~~diaper~~ manufacturer's requirements.

Where, for example, sphagnum is bleached, it can also be protonated if necessary.

### Series 3

Infill material from a diaper were protonated and tested as before, but in this instance using hydrochloric acid and using fresh urine 27g or 250ml excreted by a two year old child. A fresh sample of protonated wood pulp weighted 5g.

As before the pH of the urine should be maintained below about 5.5 (to prevent growth of bacteria) until a soiled diaper would be changed, for example up to ten hours. It should be noted that at urine pH of about 6.2 there are buffers present which mitigate the acidic effect of the infill material, and although such a urine pH is not usual, infill material of stronger pH would have to be used. The results of these experiments are shown in Table III. Series 4 of experiments are shown in Table IV.



TABLE 1.

Incubation Period at 36.9 C.										Date: 4:9:1988.
pH. of Urine	Type of Sample.	pH. at 10.00hrs.	pH. at 12.00hrs.	pH. at 13.00hrs.	pH. at 14.00hrs.	pH. at 15.00hrs.	pH. at 16.00hrs.	pH. at 17.00hrs.		
5.6	WOOD PULP.	5.3	5.3 No NH <sub>3</sub> .	5.3 No NH <sub>3</sub> .	5.3 No NH <sub>3</sub> .	5.3 No NH <sub>3</sub> .	5.3 NH <sub>3</sub> found.	5.4 NH <sub>3</sub> found.		
	SPHAGNUM MOSS.	3.8	3.8	3.8	3.8	3.8	3.8	3.8		
			No NH <sub>3</sub> . was found in any of these Samples.							
	PEAT.	3.3	3.3	3.3	3.3	3.3	3.3	3.3		
No NH <sub>3</sub> . was found in any of these Samples.										

TABLE 2.

Incubation Period at 36.9 C.										Date: 13/17:0:1988
pH. of Urine	Type of Sample.	pH. at 13.30hrs.	pH. at 14.15hrs.	pH. at 16.30hrs.	pH. at 20.30hrs.	pH. at *07.30hrs.	pH. at *16.00hrs.	* Four day tests on 17:8:1988.		
6.4	WOOD PULP.	6.6	6.6 NH <sub>3</sub> found.	6.8 NH <sub>3</sub> found.	7.2 NH <sub>3</sub> found.	7.8 NH <sub>3</sub> found.	7.8 NH <sub>3</sub> found.			
	SPHAGNUM MOSS.	4.2	4.2	4.2	4.2	4.2	4.2			
		No NH <sub>3</sub> . was found in any of these Samples.								
	PEAT.	4.0	4.0	4.0	4.0	4.0	4.0			
No NH <sub>3</sub> . was found in any of these Samples.										

Table III

Initial Urine pH	6.4	6.4	5.5.	5.5	5.6	5.6
Treated Wood Pulp pH	2.0	4.0	3.5	4.2	2.6	3.1
Final Urine pH	3.8	7.0	5.0	5.3	4.2	4.9
Remarks	no NH <sub>3</sub> found after 12 Hrs	trace NH <sub>3</sub> found after 3hrs and a further increase after 12 hrs	no NH <sub>3</sub> found after 12hrs.	insignificant trace of NH <sub>3</sub> after 6 hrs.	no NH <sub>3</sub> found after 12 Hrs.	no NH <sub>3</sub> found after 12 hrs.

As can be seen in almost all samples, no significant amount of NH<sub>3</sub> was found, and even when the pH of the urine is about 6.2, this can be sufficiently reduced using acidic infill material preferably stronger than about pH<sub>3</sub>, although it has been found by experimentation that a pH of about 2.54 is best.

SERIES 4 OF EXPERIMENTS CARRIED OUT TO SHOW THE TIME LAG  
BEFORE AMMONIA STARTS TO FORM.

TABLE IV.

EXPERIMENTS COMMENCED AT 10.00 HOURS ON 4:9:1988.  
ENDED AT 19.00 HOURS ON 4:9:1988.

URINE USED WAS pH.5.6.

VARIOUS TYPES OF ACID WERE USED

WEIGHT OF SAMPLE USED WAS 5.00 GRAMS WITH 27.00 GRAMS OF URINE IN EACH CASE.

[illegible]

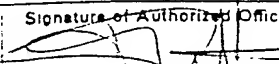
Claims

1. In a diaper, incontinent bedding and the like, the absorbent material of which is capable of releasing  
5 hydrogen ions to maintain the pH of urine in contact therewith, from 2 to 5.5 for a practicable period.
2. A diaper, incontinent bedding and the like as claimed in claim 1 wherein the absorbent material is a polymer of glucose, the free hydroxyl groups carrying the  
10 hydrogen ions.
3. A diaper, or incontinent bedding and the like, as claimed in claim 2 wherein an absorbent material was protonated to form the acidic absorbent material.
4. A diaper, or incontinent bedding and the like, as  
15 claimed in claims 1 or 2 wherein the absorbent material is naturally acidic.
5. A diaper, or incontinent bedding and the like, as claimed in claim 4 wherein the absorbent material is sterilised sphagnum moss or peat.
- 20 6. A diaper, or incontinent bedding and the like, as claimed in claim 5 wherein the sphagnum moss or peat are bleached.
7. A diaper, or incontinent bedding and the like, as claimed in any one of the preceding claims wherein the urine  
25 maintained pH is from 2.5 to 3.
8. A process of preparing an acidic absorbent material for a diaper, incontinent bedding or the like, comprising protonating an absorbent material capable of taking up hydrogen ions so that it is capable of  
30 maintaining the pH of urine in contact therewith to from 2 to 5.5 for a practicable period.
9. A process as claimed in claim 8 wherein the absorbent material includes free hydroxyl groups which take up and release the hydrogen ions.
- 35 10. A process as claimed in claim 9 wherein the absorbent material is soaked in an acid solution to protonate it.
11. A process as claimed in claim 10 wherein the acid is sulphuric or hydrochloric acid.

12. An absorbent material as defined in any one of claims 1 to 7 or made in accordance with a method of claims 7 to 11.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 89/00013

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) *		
According to International Patent Classification (IPC) or to both National Classification and IPC IPC4: A 61 L 15/00, A 61 F 13/18, A 41 B 13/02		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System	Classification Symbols	
IPC4	A 61 L; A 61 F; A 41 B	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched *		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT <sup>8</sup></b>		
Category *	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No <sup>13</sup>
X	EP, A2, 0138179 (CHEMIEFASER LENZING AKTIENGESELLSCHAFT) 24 April 1985, see claims 1-9 --	1-3,7,9-12
X	EP, A2, 0138178 (HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN) 24 April 1985, see page 2 - page 6 --	1-3,7,9-12
X	US, A, 3067745 (ALFRED A. BURGENI AND JOHN C. SIMKEVICH) 11 December 1962, see the whole document --	1-3,7,9-12
X	US, A, 3580253 (LEO J. BERNARDIN) 25 May 1971, see the whole document --	1-3,7,9-12
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>* Special categories of cited documents: <sup>10</sup></p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&amp;" document member of the same patent family</p> </div> </div>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search 5th April 1989	Date of Mailing of this International Search Report <b>14 APR 1989</b>	
International Searching Authority <b>EUROPEAN PATENT OFFICE</b>	Signature of Authorized Officer  <b>P. G. VAN DER PUTTEN</b>	

## III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
X	US, A, 4685909 (RONALD W. BERG ET AL) 11 August 1987, see column 8, line 42 - column 9, line 5 --	1-3,7,9- 12
X	US, A, 4215692 (YVON G. LEVESQUE) 5 August 1980, see the whole document --	1,4-6, 12
X	GB, A, 140228 (ADAM FORMAN) 25 March 1920, see the whole document --	1,4-6, 12
A	DE, A1, 2448471 (THE PROCTER & GAMBLE CO.) 24 April 1975, see the whole document -- -----	1-12

ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO.

PCT/GB 89/00013

SA 26332

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.  
The members are as contained in the European Patent Office EDP file on 03/03/89  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A2- 0138179	24/04/85	DE-A- 3337443 JP-A- 60173161 US-A- 4634439	25/04/85 06/09/85 06/01/87
EP-A2- 0138178	24/04/85	DE-A- 3337444 JP-A- 60100962 US-A- 4634438	25/04/85 04/06/85 06/01/87
US-A- 3067745	11/12/62	NONE	
US-A- 3580253	25/05/71	NONE	
US-A- 4685909	11/08/87	EP-A- 0202127 GB-A-B- 2175210 AU-D- 57415/86 JP-A- 62028402	20/11/86 26/11/86 20/11/86 06/02/87
US-A- 4215692	05/08/80	NL-A- 7901380 GB-A-B- 2015055 LU-A- 80953 FR-A-B- 2423171 JP-A- 54125897 AU-D- 44357/79 AU-A- 528358 SE-A- 7901488 CA-A- 1156001 AT-A- 374350 CH-A- 641688 SE-A-C- 444263	23/08/79 05/09/79 29/10/79 16/11/79 29/09/79 30/08/79 28/04/83 22/08/79 01/11/83 10/04/84 15/03/84 07/04/86
GB-A- 140228	25/03/20	NONE	
DE-A1- 2448471	24/04/75	NL-A- 7413519 FR-A-B- 2247172 BE-A- 821036 LU-A- 71095 AU-D- 74291/74 GB-A- 1484501 CA-A- 1022457 AT-A- 341971 JP-A- 50089159 US-A- 3964486 SE-A- 7412903	17/04/75 09/05/75 14/04/75 24/06/75 15/04/76 01/09/77 13/12/77 10/03/78 17/07/75 22/06/76 16/04/75



**PUB-NO:** WO008906142A1  
**DOCUMENT-IDENTIFIER:** WO 8906142 A1  
**TITLE:** pH CONTROLLING  
ABSORBENT PRODUCT AND  
METHOD FOR PREPARING  
THE SAME  
**PUBN-DATE:** July 13, 1989

**INVENTOR-INFORMATION:**

NAME	COUNTRY
FORDE, LIAM PATRICK	GB

**ASSIGNEE-INFORMATION:**

NAME	COUNTRY
FORDE LIAM PATRICK	GB

**APPL-NO:** GB08900013

**APPL-DATE:** January 3, 1989

**PRIORITY-DATA:** GB08800093A (January 5, 1988)

**INT-CL (IPC):** A61L015/00 , A61F013/18 , A41B013/02

**EUR-CL (EPC):** A61F013/15 , A61L015/40 , A61L015/46

**US-CL-CURRENT:** 604/360

**ABSTRACT:**

In a diaper the absorbent material is acidic and capable of releasing  $H^{+}$  ions to lower the pH of urine in contact therewith to below that in which bacteria producing ammonia can grow (about 5.5). The material can be naturally acidic such as sphagnum moss or peat but preferably a neutral absorbent material is protonated.